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A PRELIMINARY STUDY OF CRACK INITIATION IN SLOW
STRAIN-RATE STRESS CORROSION CRACKING OF LINEPIPE STEEL "AE"

G.R. HOEY and R.R. RAMSINGH
CORROSION SCIENCE SECTION

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A PRELIMINARY STUDY OF CRACK INITIATION IN SLOW STRAIN-RATE STRESS
CORROSION CRACKING OF LINEPIPE STEEL "AE"

by

G.R. Hoey* and R.R. Ramsingh**

ABSTRACT

Crack initiation and propagation during slow strain-rate stress corrosion cracking of linepipe steel "AE" in a CO_3/HCO_3 environment were determined by a metallographic method. Results are reported and their significance discussed. Improvements in the experimental method for determining crack velocity were outlined.

*Research scientist and **technologist, Physical Metallurgy Research Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa.

LABORATOIRES DE RECHERCHE EN MÉTALLURGIE PHYSIQUE
RAPPORT ERP/PMRL 83-1(TR)

UNE ÉTUDE PRÉLIMINAIRE SUR L'INITIATION DE LA FISSURE PAR CORROSION D'UN
ACIER DE CANALISATION DE TYPE "AE" SOUMIS LENTEMENT À LA TENSION

par

G.R. Hoey* et R.R. Ramsingh**

RÉSUMÉ

On a déterminé l'initiation et la propagation de la fissure d'un acier de canalisation de type "AE" soumis lentement à la tension en milieu CO_3/HCO_3 , au moyen d'une méthode métallographique. Le présent rapport présente les résultats de cette étude ainsi que leur pertinence. On y décrit également les améliorations apportées à la méthode expérimentale pour la détermination de la vitesse des fissures.

*Chercheur scientifique et **technologue, Laboratoire de recherche en métallurgie physique, CANMET, Énergie, Mines et Ressources Canada, Ottawa.

INTRODUCTION

The slow strain rate technique (SSRT) developed mainly by Parkins over a period of 20 years has found valuable use in studies of stress corrosion cracking (SCC) of low-strength steels in carbonate-bicarbonate, nitrate and hydroxide environments (1). In fact, conventional constant load and constant strain methods with smooth tensile samples are applicable to tests in nitrate and hydroxide solutions but not to CO_3/HCO_3 , which is considered to be the main environmental factor leading to SCC failures in buried gas transmission pipelines (2). One of the parameters used to express the SCC susceptibility of linepipe steels in CO_3/HCO_3 environments is T_E/T_{Air} , where T_E and T_{Air} are the times to failure in the chemical environment and in air. This ratio can be made more exact by replacing T_E by the time from crack initiation to failure which, of course, is less than T_E and could be a function of such variables as temperature, potential and pH. The SSRT was used to investigate crack initiation and propagation in parent metal tensile samples of linepipe "AE" (3).

The object of this investigation is to develop a technique for measuring crack propagation time, that is, the time from crack initiation to failure.

EXPERIMENTAL

The electrochemical cell, SSRT equipment and tensile sample have been described previously (3). The only modification to the apparatus was to employ a Wenking potentiostat so that polarization current could be monitored. The strain rate in this study of SCC of linepipe "AE" was $2 \times 10^{-6} \text{ s}^{-1}$. Parent metal sample were cut perpendicularly to the seam weld of a length of commercial pipe, flattened as well as possible except in the gauge length area and machined to specifications. The gauge length had a diameter of 2.5 mm and length of 13 mm. The tensile sample was coated with an electrochemical stop-off lacquer apart from the gauge length area. The chemistry of the steel is presented in Table 1. Stress corrosion tests were conducted in deaerated 1N $\text{Na}_2\text{CO}_3 + 1\text{N NaHCO}_3$ solutions at 40°C. Potentials are given on the saturated calomel electrode scale (SCE).

Non-destructive examination of the tensile sample under the microscope did not reveal crack initiation because of the surface curvature. Crack

initiation and propagation were studied by interrupting the SCC test and examining cross sections of the sample gauge length metallographically. Stress corrosion cracking tests at -600 mV were interrupted before failure at 4, 8, 12, 14, 18 and 24 hours. Time-to-failure tests were conducted also at -600 mV. Load-time and current-time curves were recorded during the experiments.

RESULTS

Table 2 summarizes corrosion morphologies, maximum crack depths and crack growth velocities for the steel "AE" at -600 mV and 40°C. These data were obtained by metallographic examination. Photomicrographs for interrupted SCC tests and for a test carried to failure are presented in Fig. 1 to 6. Cracks formed along the gauge length are stress corrosion cracks. SEM studies on fracture morphology are underway.

Stress vs time data are presented in Fig. 7. Anodic polarization current vs time data are presented in Fig. 8 for a sample fractured at -600 mV. The rest potential in deaerated CO_3/HCO_3 solution was -860 mV before an SCC test was commenced. However, a floating rest potential was observed after the samples were polarized. The floating rest potential was in the range -610 to -620 mV after 12 hours of polarization at -600 mV.

A black powdery film formed on the gauge length of the samples during SCC tests at -600 mV. Siderite and a small amount of graphite were identified in the film by X-ray diffraction analysis.

DISCUSSION

Interrupted SCC tests were employed by Andresen to determine crack initiation and propagation in Type 304 stainless steel in high purity water by SSRT (4). Crack initiation measured by metallography and optical microscopy depended on the condition of the specimen surface, the degree of sensitization and the temperature. The feasibility of the method for linepipe steel in a CO_3/HCO_3 environment is demonstrated by the data presented in this report.

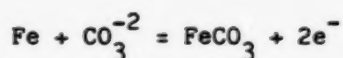
Crack initiation was associated with pits, which act as stress raisers (Fig. 1-6). The average crack propagation rate was 2×10^{-6} mm/s (Table 2). Because the stress intensities at crack tips are unknown in slow strain-rate testing of smooth tensile samples, the conclusions drawn from

propagation rates generated from this procedure are tentative. Although crack propagation rates are usually measured by constant load or constant strain tests on precracked notched samples, these tests have the disadvantage that rate data might depend on crevice corrosion; the complete absence of crevice corrosion at the crack tip seems unlikely.

Crack velocity data obtained during SCC testing of X-52 linepipe steel in a sodium hydroxide environment by SSRT have been reported in the literature (5). In a 33% sodium hydroxide solution at 92°C, crack velocities determined from measurements of crack depth and total time to failure varied in the range 0.3×10^{-6} to 2×10^{-6} mm/s depending on strain rate. Although crack initiation could occur before the yield point, Fig. 7 and Table 2 show that the velocity should be calculated from the crack depth and the difference between the SCC test fracture time and the crack initiation time. Although stress corrosion cracking velocities obtained by this method are not directly applicable in the design of structures, knowledge of relative propagation rates can be useful in comparing SCC susceptibilities of various linepipe steels.

A crack growth rate of 2×10^{-6} mm/s is equivalent to severe corrosion (6.3 cm/a). The anodic current density at the crack tip calculated from Faraday's Laws and the crack growth rate, would be 5.5 mA/cm^2 assuming the dissolution mechanism was valid for SCC of "AE" steel.

Figure 8 shows that time to passivation during SCC of "AE" steel at -600 mV was 5 hours. X-ray diffraction results indicate that passivation was caused by a film containing siderite (FeCO_3) and a small amount of graphite. Siderite is formed by the following reaction at a standard potential of -1000 mV:



Magnetite, which is believed to be instrumental in SCC of buried gas transmission pipelines, was not detected in the film by X-ray diffraction. A rapid rise in current to 0.15 mA, followed by a drop to zero current occurred when the sample fractured (Fig. 8). The formation of "fresh" metal surface and its passivation at the time of fracture would explain this pulse of current.

CONCLUDING REMARKS

Crack initiation and propagation data would be useful for comparing SCC susceptibilities of different linepipe steels.

Improvements to the SSRT would be advantageous so that measurements of crack initiation and velocity can be made on the same tensile sample without interruption of an SCC test before failure. This can be achieved by the development and use of a new stressed-electrode cell for in situ microscopy of tensile samples with a flat surface in the gauge length.

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REFERENCES

1. Parkins, R.N. "Development of strain-rate testing and its implications"; In: Stress Corrosion Cracking: The Slow Strain-Rate Technique; ASTM STP 665; 5-25; 1979.
2. Fessler, R.R. and Barlo, I.J.; In: Interpipe '79; Houston, Texas; 16-18; Jan. 1979; 197-207.
3. Revie, R.W. and Ramsingh, R.R. "Effects of potential on stress corrosion cracking of Grade 483 (X-70) HSLA linepipe steels"; Division Report ERP/PMRL 82-21(J); CANMET, Energy, Mines and Resources Canada; 1982.
4. Andresen, P.L. "Crack initiation in CERT tests on type 304 stainless steel in pure water"; Corrosion 38:1; 53-59; 1982.
5. Berry, W.E. "Stress Corrosion Cracking Laboratory Experiments"; Fifth Symposium on Linepipe Research; Houston, Texas; V-1-40, American Gas Association, Arlington, Virginia; Nov. 1974.

Table 1. Composition of steel "AE", wt %.

C	S	P	Si	Mn	Ni	Cr	Mo	V	Nb	Cu
0.08	0.005	0.011	0.29	1.98	0.033	0.097	0.24	0.05	0.045	0.053

Table 2. Crack initiation and propagation after slow strain-rate testing under potentiostatic control.

Test time h	Corrosion morphology	Crack depth mm	Propagation time h	Crack velocity mm/s
4	uniform	-	-	-
8	uniform	-	-	-
12	pits	-	-	-
14	pits & crack initiation	-	0	-
18	cracks, pits	0.035	4	2.4×10^{-6}
24	cracks, pits	0.10	10	2.7×10^{-6}
29	cracks, pits, fracture	0.10	15	1.8×10^{-6}

-600 mV, 40°C

1N Na₂CO₃ + 1N NaHCO₃

Single tests at each test time

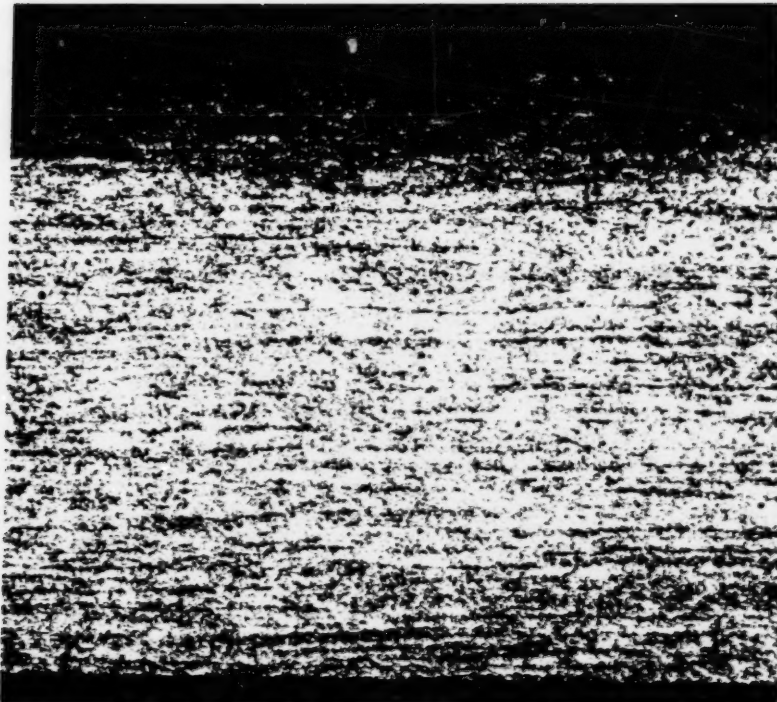


Fig. 1. Uniform corrosion of "AE" steel strained for 8 h; X50.
(neg 8649)



Fig. 2. Pitting corrosion of "AE" steel strained for 12 h;
X600. (neg 8648)

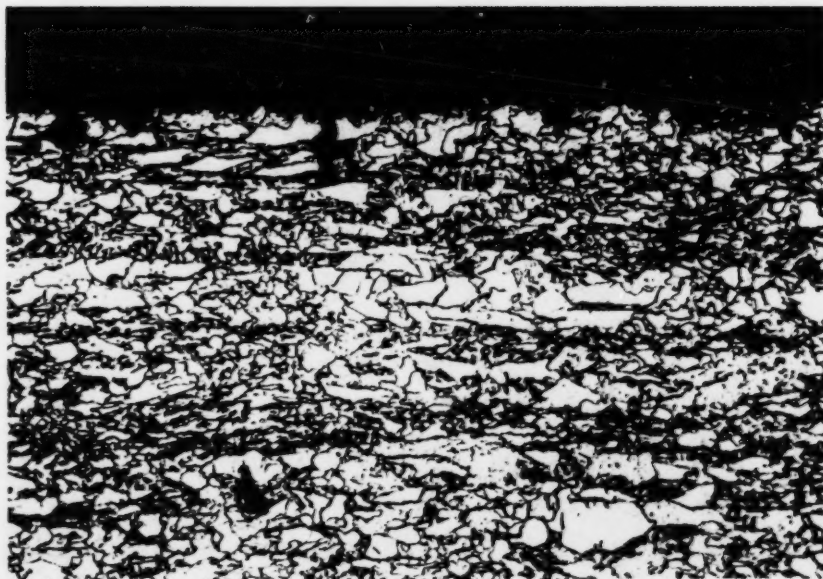


Fig. 3. Crack initiation of "AE" steel strained for 14 h;
X400. (neg 8650)

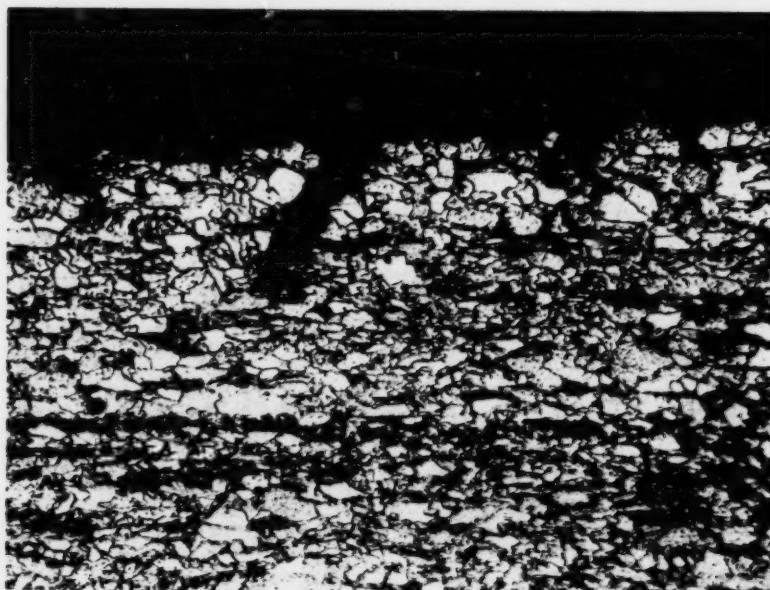


Fig. 4. Intergranular cracks of "AE" steel strained for 18 h;
X500. (neg 8651)

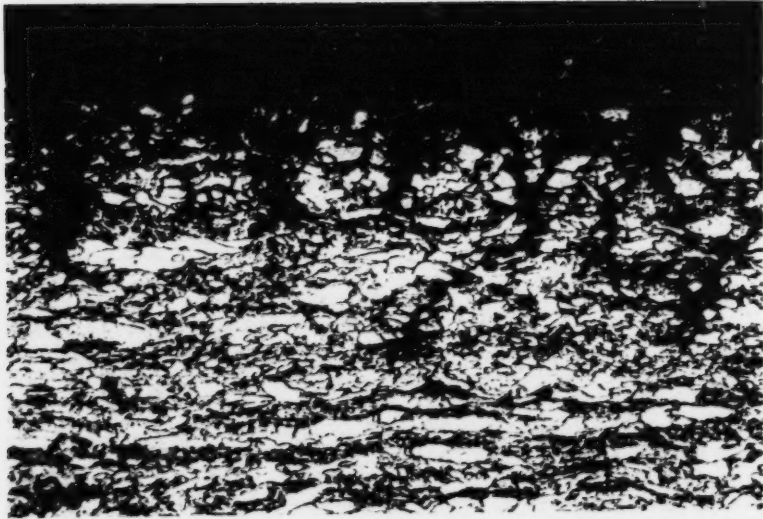


Fig. 5. Intergranular cracks of "AE" steel strained for 24 h; X300. (neg 8652)

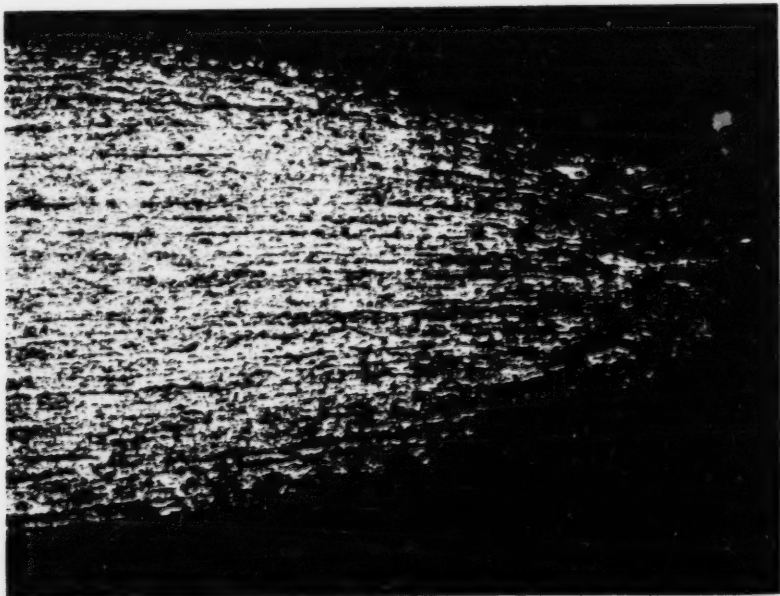


Fig. 6. Intergranular SCC of "AE" steel that failed in 29 h; X50. (neg 8653)

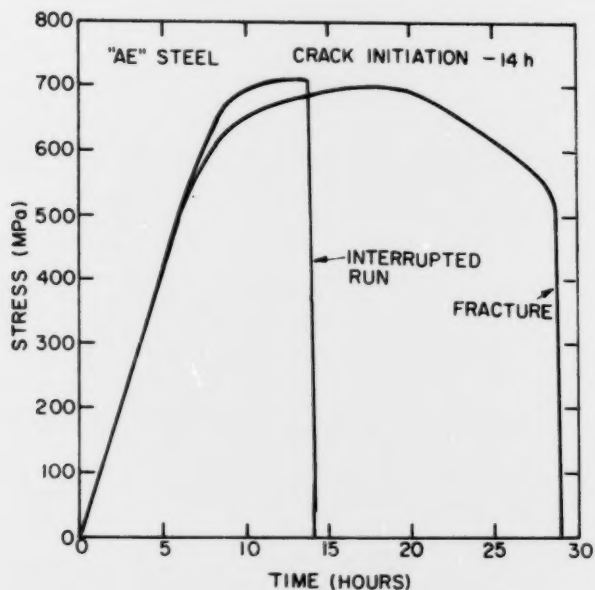


Fig. 7. Stress-time curves for "AE" steel strained in 1N Na_2CO_3 + 1 N NaHCO_3 at -600 mV and 40°C. (neg 8514)

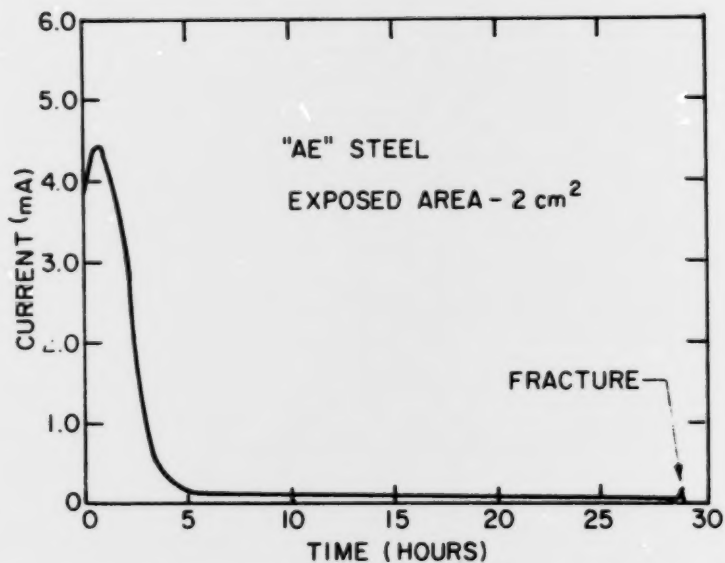


Fig. 8. Current-time curve for "AE" steel strained to failure in 1N Na_2CO_3 + 1N NaHCO_3 at -600 mV and 40°C. (neg 8515)